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Modification, ablation and hardening of metallic surfaces by a cryogenic nitrogen jet

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Abstract

This contribution gives the first results of an ongoing research aiming at developing a new surface treatment technique using a supercritical nitrogen jet, named Jazolthop, for surface modification. As nitrogen is naturally recycled within air, this new process has a high potential for surface treatment without any chemical, physical or sewage effluents. This contribution shows that, pending on the operating condition, the technique can be used (i) under a stripping or ablation mode as well as, in a "less conventional" approach, (ii) for surface hardening.

Illustration of the ablation mode is given for a Ti-6Al-4V alloy treated under static conditions using an intrusive jet. After 2 min of treatment, a thickness of 200 µm was removed from the surface by successive stripping out of micro-chips.

Illustration of the hardening mode is given through the analysis of stainless steels treated under the cryogenic jet at a moving torch velocity of 5 mm/min. The jet conditions were selected to be less intrusive and trigger the martensitic transformation without creating surface flaws of micro-cavities. In this case, the hardness of the steels was more than doubled after the passage of the cryogenic jet.

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Keywords: Surface ablation, Surface hardening, AISI 316L steel ; AISI 304L steel, Ti6Al4V

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1. Introduction

Because the service life time of industrial parts essentially depends on the surface properties (wear, fatigue and corrosion resistance for example), surface optimization treatments are more and more regarded as an essential step for the optimization of metallic parts. Thus, techniques like laser treatments [1,2], electron beam treatments [3-5] low temperature plasma nitriding [6–8] and plasma carbonitriding [9] have been attempted to improve the surface hardness or wear / corrosion balance of steels. These techniques are however energy and/or time consuming. Also, the surface cleaning or stripping techniques, that are more and more used for surface preparation and conditioning, use generally toxic chemicals or expensive laser [10] / electron [11-13] beams techniques as well as plasma reactive technologies [14]. Among the very few clean methods developed for surface modification that have recently received attention, a cryogenic nitrogen jet under high pressure has been used to strip surfaces [15]. The technology is based on the use of a generator system, named NitroJet, that has been developed by the Nitrocision LLC company (USA). The overall supercritical nitrogen jet technique itself, named Jazolthop, has been implemented and developed by the CRITT TJFU (France). It has a high potential for surface stripping while replacing complex chemical systems or water jets having sewages or toxic and harmful effluents by a jet of neutral and abundant nitrogen that can be naturally recycled in air. Despite these interesting features, little work has concentrated on the use of the Jazolthop nitrogen jet system [15, 16]. Dubs et al. [16] have explored, through numerical simulations, the effect of the standoff distance and injection conditions (nozzle pressure) on the characteristics of the expending nitrogen jet in a "free" state or when impinging onto a surface. Complementary, Laribou et al. [15] have analyzed the mechanisms of the interaction between the jet and surfaces and they have identified a large variety of surface damages responsible for the stripping process and surface removal. Depending on the materials and test parameters, the jet/surface interaction modes included cleavage, spalling, blistering, crack nucleation and growth as well as some ductile deformation. The authors also indicated that these modes could superpose in the same test and even in the area of a given sample [15].

The present contribution gives our first results of an ongoing research aiming at determining further the potential of this new technique for surface modification and the effect of the processing conditions on the conventional stripping or ablation mode for which this technique was developed as well as a "less conventional" approach for surface hardening applied solely.

2. Surface treatment technology

The pressure generator system provided by Nitrocision (USA) has been implemented by the CRITT-TJFU research center of Bar-le-Duc (France) to generate a high velocity supercritical nitrogen jet. A sketch of the overall equipment is given in Figure 1. It consists of two stages of pumps and heat exchangers that bring high pressure liquid nitrogen through a nozzle via a flexible high pressure whip authorizing the torch to be manipulated by a robot over the surface to be treated. The treatment then consists in impacting the surface with this cryogenic nitrogen jet. The pressure of the nitrogen liquid obtained by the generator system before releasing in the atmosphere through the nozzle can reach 350 MPa with an outlet temperature of about - 150°C. The treatment can be done under static condition or by dynamically moving the torch over the surface. The experimental parameters that can be modified at the level of the jet are (i) the diameter of the nozzle, (ii) the standoff distance and (iii) the jet pressure. All of these parameters have an influence on the exact nature of the thermo-mechanical jet energy as well as on its thermo-dynamic features [16]. During its flight time before impacting the surface, the jet undergoes a supersonic - subsonic transition through a Mach disc, while its pressure decreases to nearly the atmospheric pressure level [16]. The process is illustrated in Figure 2 where a torch carries the nitrogen jet over a metallic surface.

In the present contribution, two configurations of treatment will be used to illustrate (i) the ablation mode under static conditions and (ii) the hardening mode under dynamic conditions. The analysis of the efficiency of the technique to remove layers under static conditions will be carried out on a Ti alloy, the well known Ti-6Al-4V alloy, by increasing the treatment dwell time (from 5 to 240 s) for the following conditions : diameter of the nozzle : 0.45 mm, standoff distance : 60 mm and pressure of the jet : 2000 bars. While under the static condition a fixed point of the surface is targeted, the potential of the hardening mode requires necessarily the transit of the jet over the surface. This dynamic action corresponds, in fact, to a configuration of treatment having a higher technological potential for industrial applications. The analysis of the hardening mode will be illustrated by experiments carried out on the 304L and 316L austenitic stainless steels. In these cases, to make sure that the jet conditions were much less intrusive, the selection of experimental parameters was less drastic : the jet pressure was reduced to 500 bars.

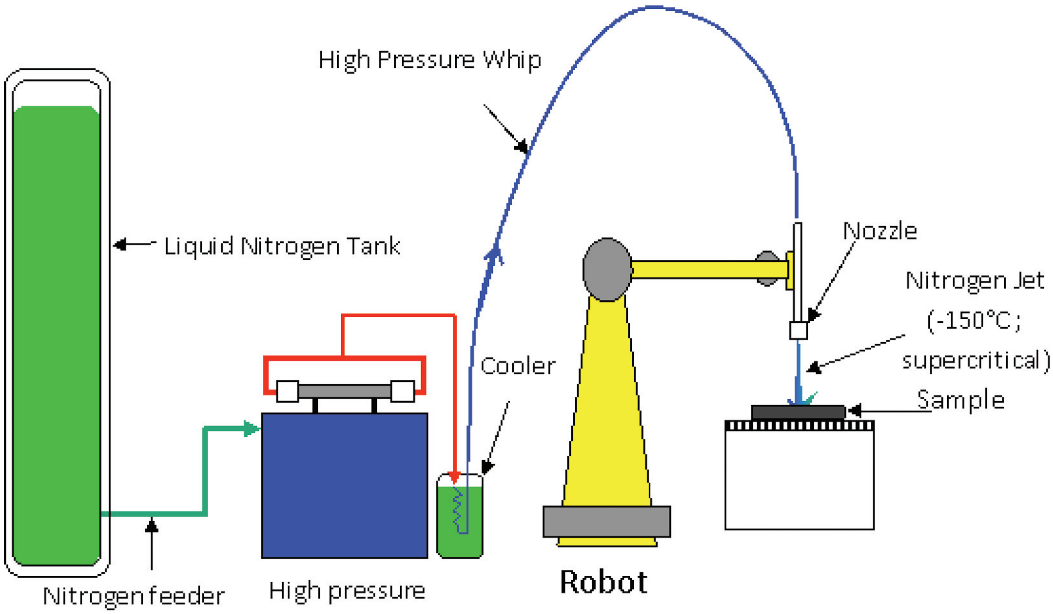

Fig. 1 :Sketch of the Jazolthop based cryogenic nitrogen jet surface treatment equipment developed in CRITT - TJFU.

Fig. 2: Image of the torch carrying the cryogenic supercritical nitrogen jet impinging on the surface of a sample.

3. Results and discussion

3.1. Stripping mode illustrated under static treatment conditions.

Figure 3 illustrates the evolution of the surface aspect, determined by 3D imaging microscopy, of the Ti-6Al-4V surface with increasing the dwell residence time of the jet at the surface. Under the jet conditions used here, a crater starts forming fairly rapidly (after 15s) at the surface and its depth increases with the treatment time.

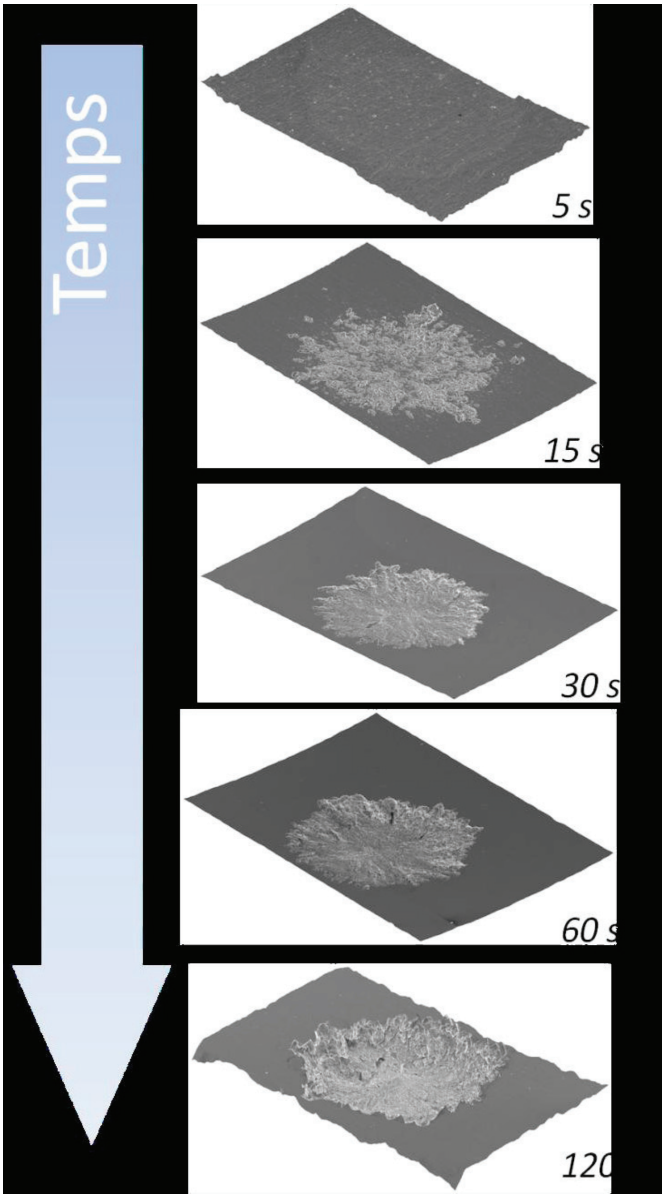

Fig. 3: Evolution of the aspect of the surface with the static treatment time for the Ti-6Al-4V alloy treated by a cryogenic nitrogen jet using the following parameters : dwell times from 5 to 120 s, nozzle diameter : 0.45 mm, standoff distance : 60 mm and jet pressure : 2000 bars.

It is interesting to notice that the width of the crater remains fairly constant over the treatment. Under the 2000 bars pressure at a standoff distance of 60 mm, a nozzle having a diameter of 0.45 mm creates a crater having a diameter slightly above 600 µm. Under these experimental conditions, the crater has a fairly flat shape. Figure 4 shows the evolution of the crater depth with the treatment time. Two regimes are observed. After a fairly linear increase up to about 60 s, where the increase in depth with time remains constant (reaching about 120 µm after 60s), the curve starts to level off. Then, for a given increase in depth, a longer treatment time is required. After 240 s of treatment a 200 µm deep hole has been created.

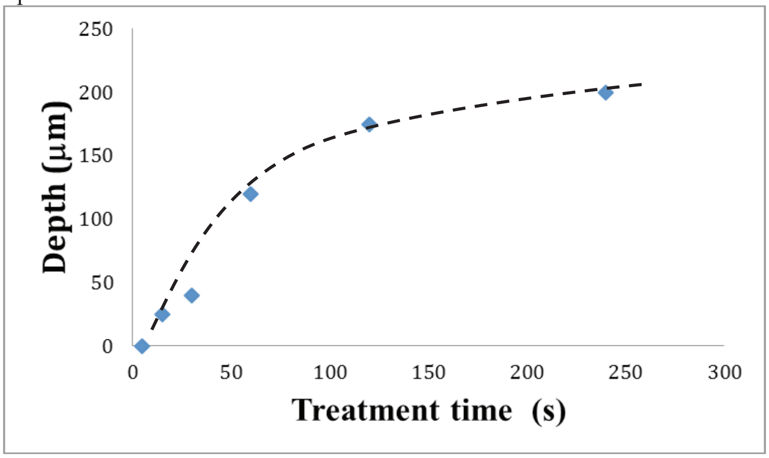

Fig. 4: Evolution of the depth of the crater with treatment time created by material stripping at the surface of a Ti-6Al-4V alloy treated by a cryogenic nitrogen jet using the following parameters : nozzle diameter : 0.45 mm, standoff distance : 60 mm and jet pressure : 2000 bars.

The aspect of the crater is detailed after 30 s of treatment by the two SEM images given in Figure 5. The general view (left) shows a fairly regular 40 µm deep carter. The higher magnification micrograph (right) taken on the edge of the crater clearly reveals that the hole was dug by successive removal and stripping of matters. Lifting of chips, having a size of about 20 µm, is observed. Comparatively to the analysis done by Laribou et al. [15] on Cu and Al based metals, it is interesting to notice that vents witnessing the passage of confine gas through the material were hardly observed in this Ti alloy under our treatment conditions.

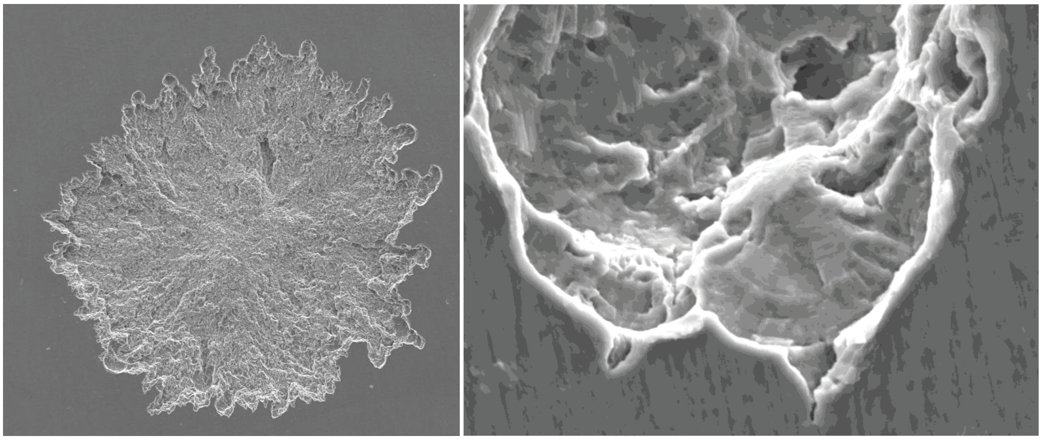

Fig. 5:SEM micrographs of the TI-6Al-4V alloy surface after 30s of static treatment. Note the rather regular shape of the 40 µm deep flat crater having a diameter of about 600 µm (left) and the successive removal of 20 µm large chips (right) on the edge of the crater.

3.2. Hardening mode illustrated by dynamic treatments.

Figure 6 gives an optical profilometer view obtained from the surface of a 316L stainless steel after the action of the nitrogen jet under successive dynamic and static treatment conditions. The nozzle used in this case had an oblong section (and not a circular one). The elongated trace visible at the right of the image witnesses the passage of the torch under dynamic condition at a velocity of 5mm/min. Analysis have revealed that the rather limited difference in height at this location compared to the untreated material is due to the formation of martensite. Hardness measurements carried out within the trace have revealed that the hardness was increased from an initial 190 Hv for the base material to about 370 Hv after the passage of the nitrogen jet. However, the trace is fairly uniform and does reveal structural damages. Comparatively, the left of the image witnesses the effect of the static jet that remained focused on the same location of the material for 10 min. As the jet conditions are much less intrusive than in the case of the Ti-6Al-4V alloy treated previously (Figures 3 to 5), the carter has reached a maximum depth of 40 μm after 10 min of jet action. The same type of observations were made using a 304L stainless steel. In the case of the 304L stainless steel however, because of the higher degree of metastability of its austenitic phase, the amount of martensite that has formed under dynamic treatment condition was higher. Figure 7 shows an optical micrograph recorded, under polarized imaging condition, at the surface of the 304L stainless steel in the vicinity of the border of the trace. The elongated lines present within the 20 mm large grains are slip traces and martensitic variants that witness the plastic deformation and stress induced martensitic transformation.

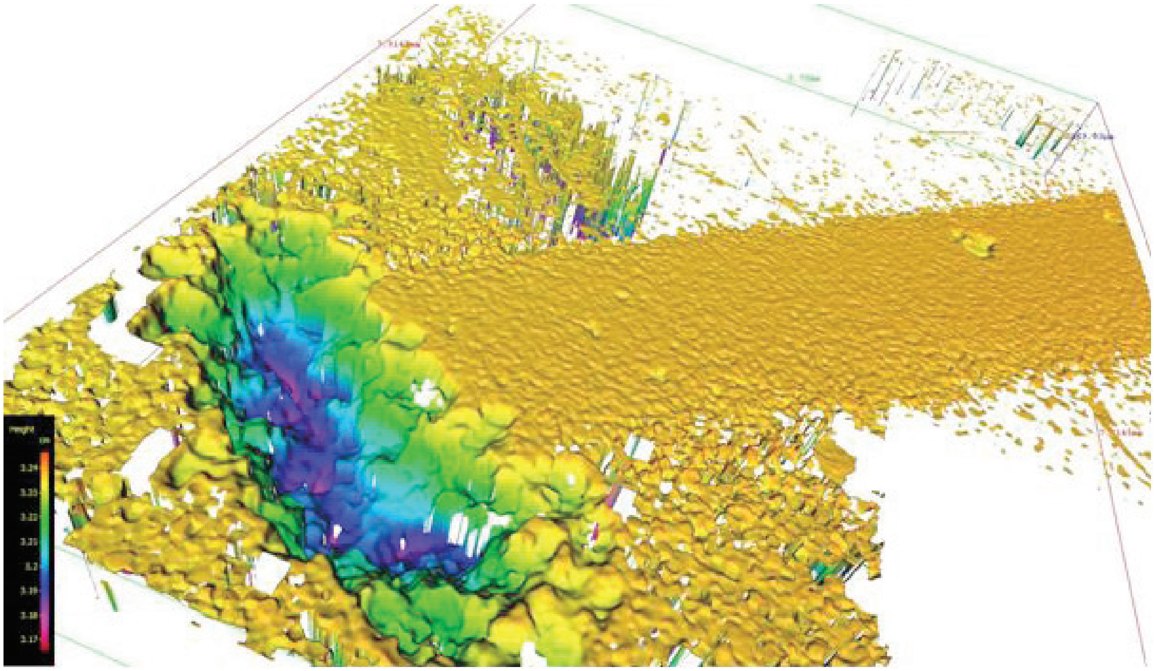

Fig. 6: profilometer view obtained from the surface of a 316L stainless steel after the action of the nitrogen jet under successive dynamic (5 mm/min) and static (10 min) treatment conditions..

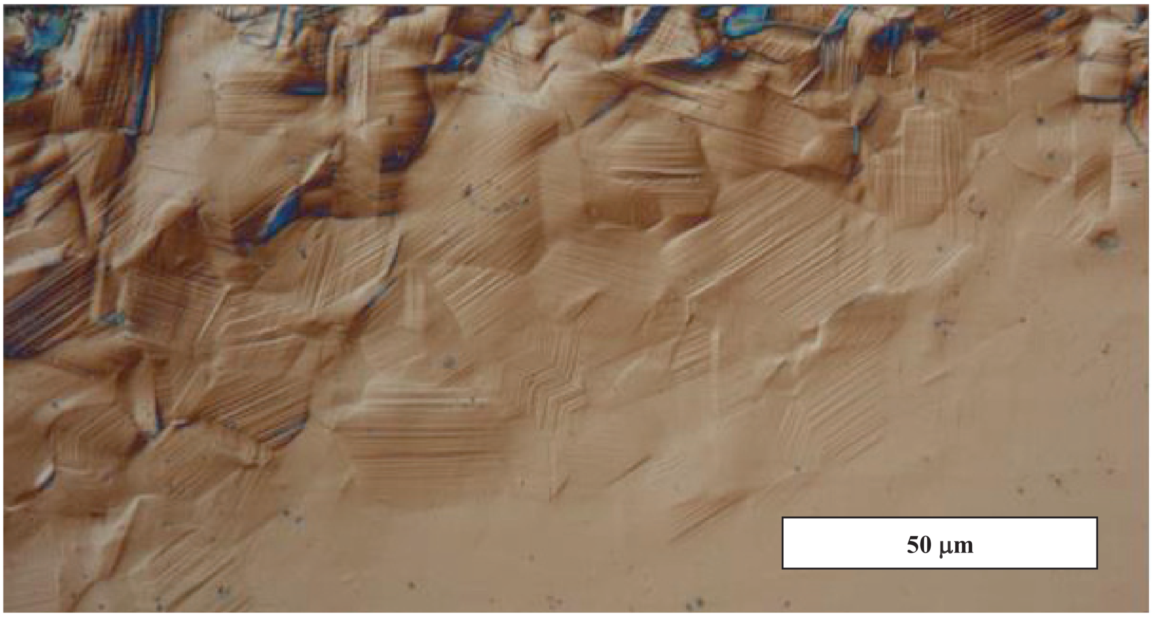

Fig. 7: Optical micrograph of the surface of a 304L austenitic stainless steel showing martensitic variants with the 20 μm austenitic parent grains. The photograph was taken at the border of the zone affected by the nitrogen jet.

Summary and conclusion

As metallic parts often fail from crack propagation initiated at their surfaces, much work is being devoted to surface modification and surface metallurgical improvement instead of treating the overall bulk material. In collaboration with "CRITT Jet Fluide", we are developing a unique treatment (no equivalent in Europe) which uses a nitrogen jet impacting the surface at cryogenic temperature to improve the material surface performances. While the use of this new technology has been establish initially for surface stripping [15], the present results show that a good setting of the experimental parameters can be done to carry out non intrusive treatments authorizing a surface hardening without formation of surface flaws or micro-cavities. Under the ablation mode, a Ti-6Al-4V alloy has been treated using a fixed intrusive jet. After 2 min of treatment, a thickness of 200 μm was removed from the surface by successive stripping out of micro-chips. Illustration of the hardening mode was given through the analysis of stainless steels. The jet conditions were selected to be less intrusive to avoid surface flaws of micro-cavities. In this case, the hardness of the steels was more than doubled after the passage of the cryogenic jet and a stress induced martensitic transformation was triggered. Further work is now under way to determine exactly the effect of each processing parameter in order to use more efficiently this new technology under the hardening or ablation modes.

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